REMARKS/ARGUMENTS

Claims 8-10, 13 and 14 have been canceled. Claims 1-7 and new Claims 15-21 are active in the case. Reconsideration is respectfully requested.

The present invention relates to a process for producing flexible polyurethane foams from polyether alcohols based on renewable materials.

Specification Amendments

The specification has been amended in order to introduce therein appropriate section headings. Entry of the amendments is respectfully requested.

Claim Amendments

Claim 1 has been amended to make a minor change thereto. No amendments have been made to Claims 2-7. Support for new Claims 15 and 16 can be found in the last paragraph of page 7. Previously active Claims 8-10, 13 and 14 are reproduced in new Claims 17-21. Entry of the amendments and new claims into the record is respectfully requested.

Claim Objection and Rejection, 35 USC 112

A copy of the standard ageing procedure for a material is provided with the present response, as well as an English translation thereof. Withdrawal of the objection to a rejection of Claim 7 is respectfully requested.

Claim Rejection, 35 USC 103

Claims 1-10, 13 and 14 stand rejected based on 35 USC 103(a) as obvious over Sugiyama et al, U. S. Patent 6,313,060. This ground of rejection is respectfully traversed.

Important aspects of the Sugiyama et al patent are directed to a particular catalyst which is identified as a double metal cyanide complex catalyst which has an organic ligand of formula (1) and a method of producing a polyether polyol by reacting an alkylene oxide and a polyhydroxyl compound that has from 2 to 8 hydroxyl groups. Polyols that are used in the preparation of polyether polyols are disclosed in column 8 of the patent. These polyols include diol, triol and sugar materials. Thus the particular metal cyanide complex that is prepared by the method shown in the patent is used to facilitate what the reference terms as a ring-opening polymerization reaction between an alkylene oxide and a polyether polyol.

Another aspect of the invention of the patent is a method of producing a resilient polyurethane foam by the reaction of the polyether polyol, that as described in the patent, with a polyisocyanate, as described in column 10 of the patent. Absolutely nowhere shown or suggested is the preparation of a polyurethane foam by the reaction of a polyisocyanate, under foam forming conditions, with a polyether polyol that is formed by the reaction of an alkylene oxide with what is termed in the present invention as a renewable material. These renewable materials are set forth in present claim 1. In this regard, it should be observed as discussed on pages 1 and 2 of the present text, that the use of renewable materials for foams that are based on renewable materials present problems. A polyol of particular importance is the reaction of castor oil and an alkylene oxide to form a flexible foam material. However, when a foamed product is obtained from castor oil, the product has been found to be unacceptable because of its odor, emissions therefrom and fogging. The product obtained displays high VOC (volatile organic compounds) and FOG (emissions of condensable compounds) values on thermodesorption, and as such are above the maximum limits that are acceptable for VOC and FOG in these foamed products. On the other hand, not only is there not so much as a hint of reacting a polyisocyanate with such a polyol that is derived from a renewable material in the patent, but there is no discussion whatever of the problems that

have been encountered in earlier attempts to form an acceptable foamed product by the reaction of a polyisocyanate with a polyol that is based on a renewable material of the types set forth on page 1 of the present specification. Accordingly, Sugiyama et al fails to suggest the present invention.

Applicants believe that the '342 reference does not overcome or improve upon the deficiencies of Sugiyama et al. The abstract of the '342 reference mentions that the polyether-polyols described therein are useful in the preparation of lubricants and polyurethanes. However, most of the disclosure of the reference, as is evident from the full text English translation of the document, is concerned with the method by which the ring-opening addition reaction occurs between an alkylene oxide and a castor oil compound in the presence of a DMC catalyst. Only the disclosure of paragraph [0028] mentions the term polyurethane, and here the reaction of the polyethers of the reference are useful as a raw material in the preparation of a polyurethane. There is no suggestion that a polyetherpolyol that is prepared from a renewable material using a DMC catalyst results in a foamed polyurethane product have acceptable odor, emission and fogging properties. Accordingly, the combined references are believed not to suggest the present invention and withdrawal of the rejection is respectfully requested.

It is now believed that the application is in proper condition for allowance. Early notice to this effect is earnestly solicited.

Respectfully submitted,

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(54) **PRODUCTION OF POLYETHER**

(57)Abstract:
PURPOSE: To obtain a polyether of a controllable molecular weight and a controllable molecular structure by performing the ring opening addition reaction of a castor oil compound and a monoepoxide as an initiator in the persence of a compound metal cyanide complex catalyst.
CONSTITUTION: A polyether is produced by effecting the ring opening addition reaction of castor oil and/or modified castor oil with a 2C or higher monoepoxide as an initiator (e.g. ethylene oxide or propylene oxide) in the presence of a compound metal cyanide complex, desirably a complex having a structure of the formula, wherein M1 is Zn(II) or 2 like; M2 is Fe (II or III), Co (II or III) or the like; R is an organic ligand (e.g. ketone, ether, aldehyde, ester, alcohol or amide), and a, b, c, d, x and y are positive integers. According to the above process, a polyether of a freely controllable molecular weight and a freely controllable structure, for example, of an Mn of 2000-50000 and an Mw/Mn of 1.3 or above can be obtained.

CLAIMS

[Claim(s)]
[Claim 1]The manufacture approach of the polyethers characterized by carrying out the ring-opening addition reaction of the with a carbon numbers of two or more mono-epoxide by making castor oil and/or denaturation castor oil into an initiator under existence of a compound metallocyanide complex compound catalyst.

[Claim 2]The manufacture approach of the polyethers of claim 1 that the number average molecular weight of the polyethers obtained is 2000-50000.
[Claim 3]The manufacture approach of the polyethers of claim 1 that the weight average molecular weight/number average molecular weight of the polyethers obtained (Mw/Mn) are 1.3 or less.
[Claim 4]The manufacture approach of the polyethers of claim 1 that mono-epoxide is alkylene oxide.
[Claim 5]The manufacture approach of the polyethers of claim 4 that alkylene oxide is what is chosen from ethylene oxide and propylene oxide.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application]This invention relates to the manufacture approach of polyether polyol especially about the manufacture approach of polyethers.

[0002]
[Description of the Prior Art]Polyethers, such as polyoxyalkylene polyol which is made to carry out the ring opening reaction of the mono-epoxide, such as alkylene oxide, to an initiator, and is obtained, are widely used for the application of the raw material of synthetic resin, such as polyurethane, a surfactant, lubricant, and others. An initiator is an active hydrogen content compound expressed with A-(H) n (A: the residue except the hydrogen atom of an active hydrogen content compound, n:1 or more integers).
[0003]As an initiator, the compound which has hydroxy groups, such as monohydric alcohol, polyhydric alcohol, a univalent phenol, and a polyhydric phenol, for example, and the compounds (alkanolamines, amines-alkylene oxide addition product, etc.) which have a hydroxy alkylamino radical are mentioned. Moreover, the polyethers which mono-epoxide is made to react to the above-mentioned initiator, and are obtained are used as an initiator. Furthermore, the attempt using denaturation castor oil, such as castor oil and ester denaturation castor oil, as an initiator is also made.
[0004]Polyethers are the following compounds which are made to carry out the ring-opening addition reaction of the mono-epoxide to the above-mentioned initiator, and are obtained.
A[-(R-O) m-H] nA: Residue R-O except the hydrogen atom of an active-hydrogen content compound: The unit m in which mono-epoxide carried out ring breakage, n:1 or more integers [0005]Conventionally, the method of making mono-epoxide react to an initiator is widely used as an approach of manufacturing polyethers, under the alkali catalyst existence represented by alkali metal compounds, such as a potassium hydroxide and a sodium hydroxide. Moreover, in composition of the polyethers of low molecular weight, he is BF3. An acid catalyst like etherate is used.
[0006]
[Problem(s) to be Solved by the Invention]When a castor oil system compound was

used as an initiator, since decomposition of the unsaturated bond included in a castor oil system compound or an ester bond was produced, the alkali catalyst known conventionally was not able to be used. When the acid catalyst of Lewis acid etc. was used, the amount polyethers of macromolecules were hard to be obtained.

[0007]

[Means for Solving the Problem]This invention is the manufacture approach of the polyethers of the amount of comparison-macromolecules which was made that the above-mentioned trouble should be solved and made especially the castor oil system compound the initiator. That is, it is the manufacture approach of the polyethers characterized by carrying out the ring-opening addition reaction of the with a carbon numbers of two or more mono-epoxide by making castor oil and/or denaturation castor oil into an initiator under existence of a compound metallocyanide complex compound catalyst.

[0008]The castor oil and denaturation castor oil which are used by this invention are denaturation castor oil obtained by processing an ester exchange reaction, a hydrogenation reaction, a sodium reduction reaction, etc. in the natural oil and this natural oil which are obtained from the seed of a castor seed.

[0009]Castor oil is partial saturation ester which consists of a glyceride of the partial saturation acid which uses a ricinoleic acid as a principal component, and has a hydroxyl group. It is possible to control the number of functional groups freely by an ester exchange reaction etc.

[0010]Especially the with a carbon numbers of two or more mono-epoxide made to react to castor oil and/or denaturation castor oil in this invention has with a carbon numbers of three or more desirable alkylene oxide. Still more preferably, the alkylene oxide of the carbon numbers 3-4, such as ethylene oxide, propylene oxide, 1, 2-butylene oxide, 2, 3-butylene oxide, and epichlorohydrin, is desirable, and is ethylene oxide and propylene oxide most preferably.

[0011]These independent mono-epoxide of others, such as those two or more sorts or they and styrene oxide, glycidyl ether, and glycidyl ester, can be used together and used. In use of two or more sorts of alkylene oxide, or use of alkylene oxide and other mono-epoxide, they can be mixed and added, or sequential addition can be carried out, and a random polymerization chain and a block polymerization chain can be formed.

[0012]Manufacturing polyethers using the compound metallocyanide complex of this invention is known (USP.3278457, USP.3278458, USP.3278459 specification). This catalyst has little generation of partial saturation mono-oar, and it is also possible to manufacture the polyethers of the amount of macromolecules extremely.

[0013]It is thought that the compound metallocyanide complex in this invention has the structure of the following general formula (1) as shown in the above-mentioned well-known example.

M1a [M2x(CN) y] b(H2 O) c Rd -- (1)

However, M1 Zn (II), Fe (II), Fe (III), Co (II), nickel (II), aluminum (III) and Sr (II), Mn (II), Cr (III), Cu (II), Sn (II), Pb (II), Mo (IV), Mo (VI), It is W (IV), W (VI), etc. and is M2. Fe (II), Fe (III), Co (II), Co (III) and Cr (II), Cr (III), Mn (II), It is Mn (III), nickel (II), V (IV), V (V), etc., R is an organic ligand, a, bx, and y are positive integers which change with a metaled valence and the metaled coordination number, and c and d are positive numbers which change with the coordination number to a metal.

[0014]M1 in a general formula (1) Zn (II) is desirable and it is M2. Fe (II), Fe (III) and Co (II), Co (III), etc. are desirable. As an organic ligand, there are a ketone, the ether, an aldehyde, ester, alcohol, an amide, etc., for example.

[0015]The compound metallocyanide complex expressed with a general formula (1) is metal salt M1 Xa (anion in which, as for M1 and a, X forms M1 and salt like ****), and poly cyano meta rate (salt) Ze [M2x(CN) y] f (M2, x, and y are the

same as that of ****.). Z is hydrogen, alkali metal, alkaline earth metal, etc. e and f are Z and M2. After mixing the solution of the mixed solvent of each water solution or water of the positive integer decided by the valence and the coordination number, and an organic solvent and contacting an organic ligand R to the obtained compound metallocyanide, it is manufactured by removing an excessive solvent and an excessive organic ligand R.

[0016]The poly cyano meta rate Ze (salt) [M2x(CN) y] f Although various metals including hydrogen or alkali metal can be used for Z, lithium salt, sodium salt, potassium salt, magnesium salt, and a calcium salt are desirable. It is the especially desirable usual alkali-metal salt, i.e., sodium salt and potassium salt.

[0017]The polyethers which have a hydroxyl group are used with the poly isocyanate compound as a raw material for polyurethane manufacture. The polyethers which have this hydroxyl group add mono-epoxide, especially alkylene oxide to the initiator which has a polyhydroxy compound, an amine compound, and other at least one active hydrogen, and it is manufactured.

[0018]In this addition reaction, alkali-metal hydroxides, such as a potassium hydroxide and a sodium hydroxide, are most ordinarily used as a catalyst. Use is also proposed for catalysts, such as 3 boron fluoride and tertiary amine, by the pan.

[0019]However, when the addition reaction of the mono-epoxide was carried out by making a castor oil system compound into an initiator, there were problems, like the molecular weight distribution of the obtained polyethers from which the polyethers of the amount of macromolecules will be hard to be obtained if there is a trouble that a castor oil system compound decomposes when an alkali catalyst is used, and the acid catalyst of Lewis acid etc. is used become large.

[0020]Since a compound metallocyanide complex compound catalyst is used for this invention, it can add mono-epoxide by making castor oil and /denaturation castor oil into an initiator. Moreover, control of molecular weight is possible and it is also possible to manufacture the polyethers of the amount of macromolecules comparatively.

[0021]Although the approach of using the metalloporphyrin other than the approach using a compound metallocyanide complex compound catalyst as an approach of compounding the polyethers of the amount of macromolecules comparatively, without using alkali or an acid catalyst using the usual initiator is learned (JP,61-197631,A), when polyethers are manufactured using metalloporphyrin, there is a problem of the polyethers obtained coloring.

[0022]When the polyethers which have the hydroxyl group manufactured by this invention are used as a polyurethane raw material, it may have a bad influence on the physical properties of the polyurethane reacted or generated at the time of the catalyst which remains in polyethers being polyurethane manufacture. It is desirable after polyethers manufacture to remove a residual catalyst.

[0023]In order to remove this catalyst from the polyethers using a compound metallocyanide complex compound catalyst, it is required to decompose and ionize a catalyst from alkali or an acid it not only to to process with filtration, an adsorbent, etc., but, and to remove these decomposition products, residual alkali, and a residual acid by adsorption, filtration, or extract after that.

[0024]The approach (JP,2-289617,A, JP,2-289618,A) of using together the approach (JP,59-15336,B) of decomposing with alkali metal or an alkali metal compound, the approach (JP,2-276821,A) of decomposing by the alkali-metal alcoholate, a buffer for pH or a buffer for pH, and a chelating agent as the approach of decomposition of a catalyst and removal, and decomposing and removing is found out. In order to remove a catalyst from the polyethers of this invention, the method of using a buffer for pH is suitable.

[0025]As for the number average molecular weight of the polyethers which have the hydroxyl group obtained in this invention, 2000-50000 are desirable. Especially

2000-30000 are desirable.
[0026]Moreover, as for the polyethers which have the hydroxyl group obtained in this invention, it is desirable that the value of weight average molecular weight/number average molecular weight (referred to as Mw/Mn below) is small. They are especially Mw/Mn. 1.3 or less are desirable.
[0027]Although the castor oil and denaturation castor oil of this invention have a hydroxyl group and it has a carboxyl group further by the case, if a compound metallocyanide complex compound catalyst is used, it is possible to add mono-epoxide to both this functional group. And the obtained polyethers have only a hydroxyl-group end.
[0028]The polyethers which have the hydroxyl group obtained by this invention are the most useful as polyol for polyurethane raw materials which is independent [its], or uses together with other polyols and is used. Moreover, the polyethers obtained by this invention are used also for the raw material of synthetic resin other than polyurethane, or the application of an additive. Furthermore, it can use as a lubricating oil, insulating oil, hydraulic oil, other oils, or its raw material. Furthermore, the polyethers obtained by this invention are changed into other compounds, such as an alkyl ether ghost and an acylation object, and it can be used for various applications.
[0029]
[Example]The addition reaction of alkylene oxide was performed to the initiator of the following castor oil system using each catalyst.
Initiator A: Polyol of three functional groups denaturalized and obtained in castor oil, and a hydroxyl value 160 (product made from Ito Oil Mill URIC H-30)
Initiator B: Polyol of three functional groups denaturalized and obtained in castor oil, and a hydroxyl value 90 (product made from Ito Oil Mill URIC H-57)
Initiator C: Polyol of four functional groups denaturalized and obtained in castor oil, and a hydroxyl value 320 (product made from Ito Oil Mill URIC H-91)
[0030][Example 1] 200 ppm (opposite workmanship) of zinc hexa cyano cobaltate catalysts were used for the 1000g initiator A, propylene oxide 1920g was added under 110-degree C conditions, and polyether polyol was obtained.

[0031][Example 2] 253 ppm (opposite workmanship) of zinc hexa cyano cobaltate catalysts were used for the 1000g initiator B, after processing with ammonia the polyol which the 70/30wt% mixture of propylene oxide and ethylene oxide was made to react under 105-degree C conditions, and was obtained [mixture] in 1700g, with the synthetic magnesium silicate, adsorption treatment was carried out, it filtered, and polyether polyol was obtained.
[0032][Example 3] After using 250 ppm (opposite workmanship) for the 1000g initiator C for the zinc hexa cyano cobaltate catalyst and processing with ammonia propylene oxide 1000g and the polyol which the 40/60wt% mixture of propylene oxide/ethylene oxide was made to react under conditions (3800g and 110 degrees C) continuously, and was obtained, with the synthetic magnesium silicate, adsorption treatment was carried out, it filtered, and polyether polyol was obtained.
[0033][Example 4] 2000 ppm (opposite workmanship) were taught by having made the potassium hydroxide into the catalyst at the 1000g initiator A, the same reaction as Example 1 was performed, the synthetic magnesium silicate performed adsorption treatment, and polyether polyol was obtained.
[0034][Example 5] He is BF3 to the 1000g initiator B. 2000 ppm (opposite workmanship) of etherate were taught, the same reaction as Example 2 was performed, the synthetic magnesium silicate performed adsorption treatment of a catalyst, and polyether polyol was obtained.
[0035][Example 6] 3000 ppm (opposite workmanship) were taught by having made the potassium hydroxide into the catalyst at the 1000g initiator C, the same reaction as Example 3 was performed, adsorption treatment of a catalyst was performed

using the synthetic magnesium silicate, and polyether polyol was obtained. the description of the polyether polyol obtained in Examples 1-6 -- a value is shown in Table 1.
[0036]
[Table 1]

		外　観	水酸基価	M_w ／M_n
実施例	例1	透　明	56.3	1.10
	例2	透　明	33.2	1.12
	例3	透　明	56.0	1.13
比較例	例4	茶　褐　色	82.5	1.7
	例5	黒色に着色	80.0	1.8
	例6	茶　褐　色	70.2	1.75

[0037]
[Effect of the Invention]This invention is the approach of manufacturing the polyethers which made the castor oil system compound the initiator. Control of the conventionally impossible molecular weight or structure can be performed freely.

Weich- und Hartschaumstoffe
Schnellalterungsprüfung
(ISO 2440 : 1997) Deutsche Fassung EN ISO 2440 : 1999

DIN EN ISO 2440

ICS 83.100

Ersatz für DIN 53578 : 1988-12

Flexible and rigid cellular polymeric materials –
Accelerated ageing tests (ISO 2440 : 1997);
German version EN ISO 2440 : 1999

Matériaux polymères alvéolaires souples et rigides –
Essais de vieillissement accéléré (ISO 2440 : 1997);
Version allemande EN ISO 2440 : 1999

Die Europäische Norm EN ISO 2440 : 1999 hat den Status einer Deutschen Norm.

Nationales Vorwort

Die Europäische Norm EN ISO 2440 wurde vom Technischen Komitee CEN/TC 249 "Kunststoffe" erarbeitet. Zuständig für die Deutsche Fassung ist der Arbeitsausschuß NMP 437 "Prüfung weich-elastischer Schaumstoffe" des Normenausschusses Materialprüfung (NMP) im DIN Deutsches Institut für Normung e.V.

Änderungen

Gegenüber DIN 53578 : 1988-12 wurden folgende Änderungen vorgenommen:

– ISO 2440 vollständig übernommen.

Frühere Ausgaben

DIN 53578: 1974-02, 1987-03, 1988-12

Fortsetzung 5 Seiten EN

Normenausschuß Materialprüfung (NMP) im DIN Deutsches Institut für Normung e.V.
Normenausschuß Kautschuktechnik (FAKAU) im DIN

Ref. Nr. DIN EN ISO 2440 : 2000-01
Preisgr. 06 a Vertr.-Nr. 2306

EUROPÄISCHE NORM
EUROPEAN STANDARD
NORME EUROPÉENNE

EN ISO 2440

November 1999

ICS 83.100

Deutsche Fassung

Weich- und Hartschaumstoffe

Schnellalterungsprüfung
(ISO 2440 : 1997)

Flexible and rigid cellular polymeric materials – Accelerated ageing tests (ISO 2440 : 1997)

Matériaux polymères alvéolaires souples et rigides – Essais de vieillissement accéléré (ISO 2440 : 1997)

Diese Europäische Norm wurde von CEN am 21. Juni 1998 angenommen.

Die CEN-Mitglieder sind gehalten, die CEN/CENELEC-Geschäftsordnung zu erfüllen, in der die Bedingungen festgelegt sind, unter denen dieser Europäischen Norm ohne jede Änderung der Status einer nationalen Norm zu geben ist.

Auf dem letzten Stand befindliche Listen dieser nationalen Normen mit ihren bibliographischen Angaben sind beim Zentralsekretariat oder bei jedem CEN-Mitglied auf Anfrage erhältlich.

Diese Europäische Norm besteht in drei offiziellen Fassungen (Deutsch, Englisch, Französisch). Eine Fassung in einer anderen Sprache, die von einem CEN-Mitglied in eigener Verantwortung durch Übersetzung in seine Landessprache gemacht und dem Zentralsekretariat mitgeteilt worden ist, hat den gleichen Status wie die offiziellen Fassungen.

CEN-Mitglieder sind die nationalen Normungsinstitute von Belgien, Dänemark, Deutschland, Finnland, Frankreich, Griechenland, Irland, Island, Italien, Luxemburg, Niederlande, Norwegen, Österreich, Portugal, Schweden, Schweiz, Spanien, der Tschechischen Republik und dem Vereinigten Königreich.

CEN

EUROPÄISCHES KOMITEE FÜR NORMUNG
European Committee for Standardization
Comité Européen de Normalisation

Zentralsekretariat: rue de Stassart 36, B-1050 Brüssel

Ref. Nr. EN ISO 2440 : 1999 D

Vorwort

Der Text der Internationalen Norm von ISO/TC 45 "Rubber and Rubber products" der International Organization for Standardization (ISO) wurde als Europäische Norm durch das Technische Komitee CEN/TC 249 "Kunststoffe" übernommen, dessen Sekretariat vom IBN gehalten wird.

Diese Europäische Norm muß den Status einer nationalen Norm erhalten, entweder durch Veröffentlichung eines identischen Textes oder durch Anerkennung bis Mai 2000, und etwaige entgegenstehende nationale Normen müssen bis Mai 2000 zurückgezogen werden.

Entsprechend der CEN/CENELEC-Geschäftsordnung sind die nationalen Normungsinstitute der folgenden Länder gehalten, diese Europäische Norm zu übernehmen:

Belgien, Dänemark, Deutschland, Finnland, Frankreich, Griechenland, Irland, Island, Italien, Luxemburg, Niederlande, Norwegen, Österreich, Portugal, Schweden, Schweiz, Spanien, die Tschechische Republik und das Vereinigte Königreich.

Anerkennungsnotiz

Der Text der Internationalen Norm ISO 2440 : 1997 wurde von CEN als Europäische Norm ohne irgendeine Abänderung genehmigt.

Warnung: Personen, die nach dieser Internationalen Norm arbeiten, sollten mit üblichen Laborpraktiken vertraut sein. Diese Norm kann nicht auf alle Sicherheitsprobleme hinweisen, wenn überhaupt, die in Verbindung mit ihrem Gebrauch stehen. Es liegt in der Verantwortung des Nutzers, angebrachte Sicherheits- und Gesundheitsmaßnahmen zu treffen und sicherzustellen, daß diese Maßnahmen den nationalen Sicherheitsbestimmungen entsprechen.

1 Anwendungsbereich

Diese Internationale Norm für Weich- und Hartschaumstoff legt die Laborverfahren fest, mit denen die Auswirkungen natürlicher Reaktionen wie Oxydation oder Hydrolyse durch Feuchtigkeit nachgestellt werden können. Die relevanten physikalischen Eigenschaften werden vor und nach der Durchführung der festgelegten Behandlungsverfahren bestimmt.

Gegenwärtig gibt es nur Prüfbedingungen für offenzellige Latex-, offen- und geschlossenzellige Polyurethanschäume und geschlossenzellige Polyolefinschäume. Prüfbedingungen für andere Werkstoffe werden nach den jeweiligen Erfordernissen ergänzt.

Die Auswirkung der Alterungsprüfungen auf die physikalischen Eigenschaften des Werkstoffs können untersucht werden; aber normalerweise werden jedoch entweder die Dehnungs- und Zugfestigkeitseigenschaften oder die Druckfestigkeitseigenschaften bzw. die Eindrückhärte geprüft.

Diese Versuche sagen nicht unbedingt etwas über das Verhalten beim Gebrauch oder die Alterung bei Lichteinwirkung aus.

2 Normative Verweisungen

Die folgenden normativen Dokumente enthalten Festlegungen, die durch Verweisung in diesem Text Bestandteil der vorliegenden Internationalen Norm sind. Zum Zeitpunkt der Veröffentlichung dieser Internationalen Norm waren die angegebenen Ausgaben gültig. Alle Normen unterliegen der Überarbeitung. Vertragspartner, deren Vereinbarungen auf dieser Internationalen Norm basieren, werden gebeten, die Möglichkeit zu prüfen, ob die jeweils neuesten Ausgaben der im folgenden genannten Normen angewendet werden können. Die Mitglieder von IEC und ISO führen Verzeichnisse der gegenwärtig gültigen Internationalen Normen.

ISO 471 : 1995
Rubber – Temperatures, humidities and times for conditioning and testing

3 Gerät

3.1 Für die Wärmealterung

3.1.1 Ofen mit Zwangszirkulation, der die erforderliche Temperatur mit einer Toleranz von ± 1 °C halten kann.

ANMERKUNG: Es wird empfohlen, die Temperatur mit einem Gerät vorzugsweise kontinuierlich aufzuzeichnen.

3.2 Für die Feuchtigkeitsalterung

3.2.1 Die Altersvorrichtung muß so groß sein, daß das Gesamtvolumen der Probekörper nicht mehr als 10 % des freien Luftvolumens einnimmt und außerdem müssen die Probekörper frei von Spannungen sein; sie müssen der Alterungsatmosphäre von allen Seiten frei ausgesetzt sein und dürfen nicht der Lichteinwirkung ausgesetzt sein.

3.2.2 Dampfautoklav oder ein ähnlicher Behälter, der die erforderliche Temperatur mit einer Toleranz von ± 1 °C halten kann und einem absoluten Druck von bis zu 300 kPa standhält.

3.2.3 Glasbehälter mit einem geeigneten Verschluß und ein Wasserbad oder Trockenofen zum Erwärmen des Behälters, der die erforderliche Temperatur mit einer Toleranz von ± 1 °C halten kann.

3.3 Zur Bestimmung der physikalischen Eigenschaften

Vorrichtung zur Bestimmung der physikalischen Eigenschaft, die untersucht werden soll.

4 Probekörper

4.1 Anzahl, Größe und Form

Anzahl, Größe und Form der Probekörper müssen auf die jeweils zu untersuchende Eigenschaft abgestimmt werden. Sie müssen vor der Alterungsprüfung auf die für die jeweilige Prüfung verwendeten Abmessungen gebracht werden.

4.2 Konditionierung

Der Werkstoff darf erst 72 h nach der Herstellung getestet werden, es sei denn, daß die Resultate nach 16 h oder 48 h nicht mehr als 10 % von denen nach 72 h abweichen. Die Prüfung nach 16 h oder 48 h ist gestattet, wenn in der spezifizierten Zeit die oben genannten Kriterien zufriedenstellend sind.

Vor der Prüfung sollen die Probekörper unverbogen und unverformt, mindestens 16 h in einer der folgenden Klimate nach ISO 471 konditioniert werden.

23 °C ± 2 °C, (50 ± 5) % relative Feuchte bzw.

27 °C ± 2 °C, (65 ± 5) % relative Feuchte

Dieser Zeitraum kann den letzten Teil der Wartezeit nach der Herstellung bilden.

ANMERKUNG: Für die Erzielung von Bezugswerten wird empfohlen, die Prüfung erst 7 oder mehr Tage nach der Herstellung des Schaumstoffes durchzuführen.

5 Verfahren

5.1 Allgemein

Nach dem Konditionieren ist die Prüfung der gewünschten physikalischen Eigenschaft durchzuführen, wobei die Probekörper schnell in den Zustand für die Alterungsprüfung zu bringen sind. Falls es sich bei der Prüfung um eine Zerstörungsprüfung handelt, z. B. eine Untersuchung der Zugfestigkeit, wird empfohlen, die Prüfungen am Referenz-Werkstoff und am gealterten Werkstoff gleichzeitig durchzuführen, d. h. nachdem der letztere den Alterungsbedingungen ausgesetzt worden ist.

Die Versuchsanordnung für die Alterung ist aus den folgenden Alternativen auszuwählen und sollte für den zu prüfenden Werkstoff geeignet sein. Es wird empfohlen, Werkstoffe mit unterschiedlicher chemischer Zusammensetzung nicht im gleichen Behältnis zu altern.

5.2 Trockene Wärmealterung

5.2.1 Temperaturen

Polyolefin: 70 °C

Latex: 70 °C oder 100 °C

Polyurethan: 125 °C oder 140 °C

ANMERKUNG: Die Verwendung der nichtstandardmäßigen Temperatur von 140 °C ist aus folgenden technischen Gründen erforderlich: Die Temperatur bei der Alterungsprüfung sollte so hoch wie möglich sein, um Ergebnisse innerhalb kürzester Zeit erhalten zu können; oberhalb dieser kritischen Temperatur entsprechen die Veränderungen, die bei der Prüfung von Polyurethan-Schaumstoff auftreten, nicht denen unter normalen Gebrauchsbedingungen, so daß die Alterungsprüfung nicht mehr zwischen Schaumstoffen mit unterschiedlichem Gebrauchsverhalten unterscheidet.

5.2.2 Dauer der Alterung

16 h, 22 h, 72 h, 96 h, 168 h bzw. 240 h oder ein Vielfaches von 168 h mit einer Toleranz von ± 5 %, aber nicht mehr als ± 4 h.

5.3 Feuchtigkeitsalterung

5.3.1 Feuchtigkeit

5.3.2 100 % relative Luftfeuchtigkeit oder gesättigter Dampf.

5.3.3 Temperaturen und Dauer der Alterung

Werkstoff	**Bedingungen**
Polyurethan	85 °C für 20 h oder
(alle Sorten)	105 °C für 3 h
Polyurethan	120 °C für 5 h
(nur Polyether)	
Toleranz bei der Temperatur:	± 2 °C

Toleranz bei der Dauer der Alterung ± 5 %, aber nicht mehr als ± 2 h, wobei die Dauer ab dem Zeitpunkt gemessen wird, ab dem die Luft im Behälter durch Wasserdampf oder Dampf ersetzt wird.

ANMERKUNG: Bei dieser Hydrolysebeständigkeitsprüfung ist die Verwendung der nichtstandardmäßigen Temperaturen von 105 °C und 120 °C aus folgenden technischen Gründen erforderlich: 105 °C werden verwendet, weil bei dieser Temperatur ein geschlossener Behälter erforderlich ist, wodurch die Kontrolle der Bedingungen besser ist als bei der alternativen Temperatur von 100 °C. 120 °C werden verwendet, weil bei dieser Temperatur die meisten experimentellen Nachweise gesammelt wurden, während bei der alternativen Temperatur von 125 °C nur wenige bzw. keine Nachweise vorhanden sind. Solange diese Hintergrundinformationen nicht zusammengetragen sind, gilt der Wechsel auf 125 °C als nicht möglich.

5.4 Rückkonditionierung

Nachdem sie die Versuchsanordnung für die Alterung durchlaufen haben, werden die der Feuchtigkeitsalterung unterzogenen Probekörper bei 70 °C ± 2 °C 3 h je 25 mm Dicke getrocknet, wobei die Mindestdauer 3 h beträgt. Danach werden die der Feuchtigkeitsalterung unterzogenen Probekörper unter den in Abschnitt 4.2 beschriebenen Bedingungen 3 h je 25 mm Dicke neukonditioniert. Die der trockenen Wärmealterung unterzogenen Probekörper müssen nur das Rückkonditionierungsverfahren durchlaufen.

Nach der Neukonditionierung sind die Eigenschaften der gealterten Probekörper zu untersuchen.

6 Darstellung der Ergebnisse

6.1 Berechnung

Die prozentuale Änderung bei der untersuchten Eigenschaft wird anhand der folgenden Formel dargestellt:

$$\frac{\bar{X}_a - \bar{X}_0}{\bar{X}_0} \times 100$$

wobei

$\bar{X}_0$ Durchschnittswert der Eigenschaft vor der Alterung;

$\bar{X}_a$ Durchschnittswert der Eigenschaft nach der Alterung.

6.2 Format

Der Wert der prozentualen Änderung wird notiert, danach folgen in Klammern die Versuchsbedingungen in der Reihenfolge Zeit, Temperatur und Methode.

BEISPIEL:

Wert in % (16 h, 70 °C, trockene Wärmealterung).

7 Prüfbericht

Der Prüfbericht muß folgende Informationen enthalten:

a) Einen Verweis auf diese Internationale Norm;

b) Beschreibung des Werkstoffes;

c) Angewendetes Verfahren und Versuchsbedingungen;

d) Durchschnittlicher Endwert der Eigenschaft;

e) Angabe der prozentualen Änderung bei der Eigenschaft nach Abschnitt 6;

f) Versuchsdatum;

g) Abweichungen von dieser Internationalen Norm.

DEUTSCHE NORM **January 2000**

Flexible and rigid cellular polymeric materials

Accelerated ageing tests
(ISO 2440 : 1997)
English version of DIN EN ISO 2440

DIN
EN ISO 2440

ICS 83.100

Supersedes DIN 53578, December 1988 edition.

Weich- und Hartschaumstoffe – Schnellalterungsprüfung
(ISO 2440 : 1997)

European Standard EN ISO 2440 : 1999 has the status of a DIN Standard.

National foreword

This standard has been published in accordance with a decision taken by CEN/TC 249 to adopt, without alteration, International Standard ISO 2440 as a European Standard.

The responsible German body involved in its preparation was the *Normenausschuss Materialprüfung* (Materials Testing Standards Committee), Technical Committee *Prüfung weich-elastischer Schaumstoffe.*

Amendments

DIN 53578, December 1988 edition, has been superseded by the specifications of EN ISO 2440, which is identical to ISO 2440.

Previous editions

DIN 53578: 1974-02, 1987-03, 1988-12.

EN comprises 6 pages.

Ref. No. DIN EN ISO 2440 : 2000-01
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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 2440

November 1999

ICS 83.100

English version

Flexible and rigid cellular polymeric materials
Accelerated ageing tests
(ISO 2440 : 1997)

Matériaux polymères alvéolaires souples et rigides – Essais de vieillissement accéléré (ISO 2440 : 1997)

Weich- und Hartschaumstoffe – Schnellalterungsprüfung (ISO 2440 : 1997)

This European Standard was approved by CEN on 1998-06-21.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, the Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Ref. No. EN ISO 2440 : 1999 E

Foreword

International Standard

ISO 2440 : 1997 Flexible and rigid cellular polymeric materials – Accelerated ageing tests,

which was prepared by ISO/TC 45 'Rubber and rubber products' of the International Organization for Standardization, has been adopted by Technical Committee CEN/TC 249 'Plastics', the Secretariat of which is held by IBN, as a European Standard.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, and conflicting national standards withdrawn, by May 2000 at the latest.

In accordance with the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard:

Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, the Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 2440 : 1997 was approved by CEN as a European Standard without any modification.

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies, for flexible and rigid cellular polymeric materials, laboratory procedures which are intended to imitate the effects of naturally occurring reactions such as oxidation or hydrolysis by humidity. The physical properties of interest are measured before and after the application of the specified treatments.

Test conditions are only given for open cellular latex, both open- and closed-cell polyurethane foams, and closed-cell polyolefin foams. Conditions for other materials will be added as required.

The effect of the ageing procedures on any of the physical properties of the material may be examined, but those normally tested are either the elongation and tensile properties, or the compression or indentation hardness properties.

These tests do not necessarily correlate either with service behaviour or with ageing by exposure to light.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 471:1995, *Rubber — Temperatures, humidities and times for conditioning and testing.*

3 Apparatus

3.1 For heat ageing

3.1.1 Oven, with forced circulation, capable of maintaining the required temperature to within ±1 °C.

NOTE — It is recommended that a device be used to record the temperature, preferably continuously.

3.2 For humidity ageing

3.2.1 Ageing apparatus, of such a size that the total volume of the test pieces does not exceed 10 % of the free air space, and such that the test pieces are free of strain, freely exposed to the ageing atmosphere on all sides and not exposed to light.

3.2.2 Steam autoclave or similar vessel, capable of maintaining the required temperature to within ±1 °C and of withstanding absolute pressures up to 300 kPa.

3.2.3 Glass vessel, with a suitable closure, and a **water-bath** or **drying oven** for heating the vessel, capable of maintaining the required temperature to within ±1 °C.

3.3 For physical-property measurements

Use apparatus appropriate to the measurements of the physical property to be examined.

4 Test pieces

4.1 Number, size and shape

The number of test pieces, and their size and shape, shall be appropriate to the property being examined. They shall be prepared, before ageing, to the dimensions used in the particular test.

4.2 Conditioning

Material shall not be tested less than 72 h after manufacture, unless at either 16 h or 48 h after manufacture it can be demonstrated that the mean result does not differ by more than ±10 % from those obtained after 72 h. Testing is permitted at either 16 h or 48 h if, at the specified time, the above criterion has been satisfied.

Prior to the test, the test pieces shall be conditioned, undeflected and undistorted, for at least 16 h in one of the following atmospheres as given in ISO 471:

23 °C ± 2 °C, (50 ± 5) % relative humidity;

27 °C ± 2 °C, (65 ± 5) % relative humidity.

This period can form the latter part of the period following manufacture.

NOTE — It is recommended that for reference purposes the test be performed 7 days or more after the cellular material has been manufactured.

5 Procedure

5.1 General

After conditioning, the test of the required physical property shall be performed and the test pieces shall be brought rapidly to the ageing condition. If the test to be performed is destructive, for example the examination of tensile properties, it is recommended that the tests on both reference and aged material be performed at the same time, that is after the exposure of the latter to the ageing conditions.

The ageing conditions shall be chosen from the following alternatives which should be appropriate to the material under test. It is recommended that materials differing in chemical composition should not be aged in the same enclosure.

5.2 Dry heat ageing

5.2.1 Temperatures

Polyolefin	70 °C
Latex	70 °C or 100 °C
Polyurethane	125 °C or 140 °C

NOTE — The use of the non-standard temperature of 140 °C is included for the following technical reason: the temperature of the ageing test should be as high as possible to enable results to be obtained in the minimum time, but above this critical temperature the changes which occur when polyurethane foam is tested are not those which are found in service, so that the ageing test will no longer discriminate between foams of different behaviour in service.

5.2.2 Duration of ageing

Use 16 h, 22 h, 72 h, 96 h, 168 h, 240 h or some multiple of 168 h, with a tolerance ±5 %, but not more than ±4 h.

5.3 Humidity ageing

5.3.1 Humidity

Use 100 % relative humidity or saturated steam.

5.3.2 Temperatures and duration of ageing

Material	Conditions
Polyurethane (all types)	85 °C for 20 h or 105 °C for 3 h
Polyurethane (polyether only)	120 °C for 5 h

Tolerance on temperature: ±2 °C

Tolerance on duration of ageing: ±5 % but not more than ±2 h, the time being measured from the time when the air in the vessel has been replaced by water vapour or steam.

NOTE – In this test for resistance to hydrolysis, the use of the non-standard temperatures of 105 °C and 120 °C is included for the following technical reasons: 105 °C is used because this temperature requires the use of a closed vessel so that control of the conditions is better than at the alternative of 100 °C; 120 °C is used because much experimental evidence has been accumulated at this temperature, but little or none at the alternative of 125 °C. Until these background data are collected it is not considered possible to change to 125 °C.

5.4 Reconditioning

After exposure to the ageing conditions, test pieces undergoing humidity ageing shall be dried at 70 °C ± 2 °C for 3 h per 25 mm of thickness, subject to a minimum of 3 h. The humidity-aged test pieces shall then be reconditioned in the atmosphere specified in 4.2 for 3 h per 25 mm of thickness. Dry-heat-aged test pieces shall merely undergo the reconditioning procedure.

After reconditioning, the properties of the aged test pieces shall be tested.

6 Expression of results

6.1 Calculation

The percentage change in the property being examined is given by the formula

$$\frac{\overline{X}_a - \overline{X}_0}{\overline{X}_0} \times 100$$

where

$\overline{X}_0$ is the average value of the property before ageing;

$\overline{X}_a$ is the average value of the property after ageing.

6.2 Format

The value of the percentage change shall be stated, followed by the test condition in parentheses, in order time, temperature and method.

EXAMPLE

Value % (16 h, 70 °C, dry heat).

7 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) a description of the material;

c) the procedure and conditions used;

d) the average final value of the property;

e) the percentage change in property, expressed as indicated in clause 6;

f) the date of the test;

g) any deviations from this International Standard.